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Standard Methods of Testing Sorbent Performance of Absorbents[1]

This standard is issued under the fixed designation F 716; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—Section 14 was added editorially in October 1993.

1. Scope

1.1 These methods cover the development of laboratory test data which describe the performance of absorbent materials used to remove oils and other compatible fluids from water.

1.2 *This standard should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.* (For a specific warning statement see 10.4.1.)

2. Referenced Documents

2.1 *Federal Standard:*

Fed. Std. No. 141 Paint, Varnish, Lacquer and Related Materials. Methods of Inspection, Sampling and Testing[2]

2.2 *Military Specification:*

MIL-I-631D Insulation, Electric, Synthetic Resin Composition, Nonrigid[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *absorption*—a process where the material taken up is distributed throughout the body of the absorbing material.

3.1.2 *adsorption*—a process where the material taken up is distributed over the surface of the adsorbing material.

3.1.3 *sorbent*—a material used to recover liquids through the mechanisms of absorption or adsorption, or both.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *oil, oily liquid*—substantially water-immiscible organic liquids (limited water solubility) as well as liquids containing dissolved solids. These oily materials may either float, sink, or be dispersed.

3.2.2 *Type I absorbent (loose)*—an unconsolidated, particulate material without sufficient form to be applied in single units. This type may include particulate blends with other materials, that is, adsorbents to allow use in specific applications.

3.2.3 *Type II absorbent (roll, sheet, pad, blanket, pillow, web)*—a material that has a form and strength sufficient to be lifted and handled when saturated without tearing.

3.2.4 *Type III Absorbent (booms)*—absorbent material in a form whose length substantially exceeds other dimensions. Booms are to be provided with connector units so that they may be coupled to another boom or line.

4. Summary of Method

4.1 The absorbent material is tested using established standard methods, where applicable, and specially developed tests for performance factors.

5. Significance and Use

5.1 These methods are to be used as a basis for comparison of absorbents in a consistent manner.

5.2 These methods are not useful for a comparison of absorbents with adsorbents, even though all absorbents exhibit adsorbent properties. Both types of materials have prime areas of utility.

5.3 These methods may not list all the safety and disposal options necessary for safe ultimate disposal of used sorbent material into the environment. Federal, state, and local regulatory rules must be followed.

6. Special Considerations

6.1 *Solubility*—Fluid should not dissolve the absorbent under normal recovery or handling conditions.

6.2 *Reuse*—Used absorbent materials may be recycled under certain conditions. Fluid absorbed cannot be released with squeezing. However, many of the polymeric materials will depolymerize at temperatures at or below those in a refinery pipe still. This means they are recoverable, although in a different form. Along with resource recovery, the harvestability of sorbents will become more important. Lower boiling materials may also be steam stripped from absorbents to recover both fluid and absorbent.

6.3 *Release of Volatiles*—Certain volatile materials picked up by sorbents may create a vapor hazard by increasing the

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[1] These methods are under the jurisdiction of ASTM Committee F-20 on Hazardous Substances and Oil Spill Response and are the direct responsibility of Subcommittee F20.12 on Removal.

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[2] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

exposed surface area and therefore vapor release. Depending on the material picked up, this may increase fire or health hazard (see 11.3).

6.4 *Release to an Alternative Material Such as Sand*—The tendency to release sorbed liquid to beaches, ground, clothing of personnel, etc., both as liquid and vapor can be determined on a basis of priority (see 11.5).

6.5 *Penetration*—Absorbents may be useful as sealing devices and the penetration test will allow a uniform means of determining the efficacy of a material for this use.

7. Apparatus

7.1 *Exterior Exposure Tester*, sufficient for use with Fed. Std. No. 141, Method 6152 (*optional*).

7.1.1 Actual outdoor exposure in a direction averaging the maximum exposure in a sunny area, such as Southern Florida or Arizona, is desired. Exposure should be listed in months with total sun days.

7.1.2 Actual outdoor exposure for a period of three months at a testing site should be listed.

7.2 *Tension Tester*—Commercially available tester (*optional*).[3]

7.2.1 Use of actual weights suspended from test pieces in turn suspended from a continuous reading hanging scale may be used. Shock loading may be simulated by fastening one end of a unit rigidly and attaching the maximum pickup weight to the bottom end hanging free. The weight can be raised 2.5 cm and released and also 10 cm and again released. This test should be recorded as pass or fail.

7.3 *Continuous Reading Hanging Scales*—A continuous reading hanging scale fitted with a hook or clamp should be used. For testing units, weighing sensitivity shall be ± 2%. For small samples, postal-type scales may be used (0 to 80 g).

7.4 *Top Loading Balance*, having 0.5-g resolution for samples weighing at minimum 50 g or more. An accuracy of 2% is desired.

7.5 *Measuring Tubes*—10 and 100-mL graduated measuring cylinders of regular laboratory quality.

7.6 *Paper Towels*, for removing excess liquid from the absorbent.

7.7 *Oven-Dried Mason Sand*, for use in checking rate of release. Screen analysis is desired. Optionally, known screen size silica sand can be used.

8. Test Fluids

8.1 Test fluids may be used for general testing, although the actual fluid should be used where possible.

8.2 *Samples Heavier than Water*—For samples heavier than water 1,1,1-trichloroethane (available in many retail stores as nonflammable spot remover) may be used.

8.3 *Low-Viscosity Materials*—For low-viscosity material (like gasoline), use xylene which has a higher flash point. (For safety, a solution containing 10 % methylene chloride can be added to aid in raising flash point.) Materials conforming to 8.2 have a viscosity similar to gasoline but the density difference may have to be adjusted.

8.4 *Polar Solvents*—For polar solvents, use methylisobutylketone (hexone), butylacetate, etc.

8.5 *Medium-Viscosity Fluids*—For medium-viscosity fluids, No. 2 fuel oil, diesel fuel, JP-5, Jet A-1 (turbine fuel), or light crude oil (for example, light Arabian or Iranian crude), is recommended.

8.6 *High-Viscosity Fluids*—Nondetergent or C-10 Transformer dielectric oil (specify manufacturer and viscosity) is recommended.

8.7 *Range Finding Fluids*—Ethanol (95%), acetone, ethylene glycol, cyclohexane, 2-nitropropane are suitable fluids.

9. Conditioning

9.1 Condition three absorbent test specimens at room temperature (21 to 26°C) (70 to 80°F) in a closed vessel with 2.5 cm of water in the bottom. Suspend specimens in vapor space without contacting water for not less than 24 h prior to testing. Condition specimens in the fully exposed state with no coverings or wrapping that would hinder the equilibration process.

9.2 Condition three absorbent test specimens totally immersed in water at room temperature (21 to 26°C) (70 to 80°F) in a vessel with a minimum of 10 cm of water covering the absorbent for not less than 24 h prior to testing. Condition specimens in the fully exposed state with no coverings or wrapping which would hinder the equilibration process.

9.3 *Alternative Temperatures*—If temperature conditions other than room temperature are expected to be important, then the sorbent should be conditioned and tested at the temperature of use in addition to those specified in 9.1 and 9.2.

10. Determination of Physical Characteristics of Absorbents

10.1 *Mildew*—The susceptibility of an absorbent to mildew under normal storage is described in MIL-I-631D. This optional test determines the expected shelf life under conditions which could lead to mildew. This test may be required when it is believed that this condition may exist prior to an emergency.

10.2 *Exterior Exposure*—This optional test may be performed in accordance with Fed. Std. No. 141, Method 6152. Actual exposure in area of use for one month is preferable. Long-term testing is not necessary for materials that are to be used in emergency situations. For testing purposes both absorbent and adsorbent and other parts of a fabricated unit should be tested or exposed individually and reported.

10.3 *Humidity/Water Resistance*—This test is used to predict the performance of a product stored under high humidity or one that is water-soaked prior to use.

10.3.1 Condition all absorbent test specimens at room temperature (21 to 26°C) (70 to 80°F) in a closed vessel with 2.5 cm of water in the bottom. Suspend the specimens in vapor space without contacting water for 2 days (14 days optional). At the end of the exposure period, test the specimens for oil and water absorbency. Record weights before and after this conditioning.

10.3.2 Weigh specimens that are conditioned in water by being total immersed (8.2) immediately after removal from water and again after wringing out all excess water (typical hand wringer for laundry or equivalent). Test specimens for oil

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[3] Instron Model No. TM, available from Instron Corp., 100-A Royall St., Canton, MA 02021, or equivalent.

and water sorbency following both weighings.

10.4 *Flammability*—This test should indicate either contribution to an existing fire, that is, a UL rating of 94, if desired, or a simple rating of combustible or noncombustible and flashpoint by Cleveland open-cup.

10.4.1 **Warning**—All sorbent materials, even those considered nonhazardous, take on the characteristics of the material they pick up. If the material is flammable, the used absorbent material is flammable. If the fluid picked up is toxic, then the used absorbent should be handled as a toxic substance. Basic active absorbent and simple fabricated units are adequate for this test.

11. Determination of Expected Performance

11.1 *General*:

11.1.1 Initial testing for performance can be accomplished with absorbent in the as-received condition. Conditioning as described in Section 9 may be necessary for special use or for special fluids to be absorbed. Good practice requires triplicate testing with results reported as the average.

11.1.2 For all tests, without exception, the fabricated materials or units must remain handleable (without tearing) at the maximum pickup rate reported while being held by a corner of the unit.

11.2 *Oily Material Absorption*—This test provides a simple method of comparing the absorbing material's inherent capacity to absorb various fluids as well as compare ultimate capacity to do so. This test is run with an excess of fluid.

11.2.1 *Type I Absorbent*—In a 10-mL graduated cylinder, place 2 mL of absorbent and weigh to determine the weight of absorbent. Place the 2 mL of absorbent into a 100-mL graduated cylinder. Add 50 mL of fluid to be tested, record level, then cap with a foil (aluminum or plastic)-wrapped cork or stopper. Agitate for short periods with a glass stirring rod. If at the end of 2 h there is still free liquid on top of the swollen absorbent or underneath if the fluid has a density greater than the absorbent, place a piece of 200-mesh screen (just slightly smaller than the inside diameter of the cylinder with at least four centering wires bent up at 90°) in the fluid to sit on top of the absorbent. Record the level of the swollen absorbent. If the fluid is much more dense than the absorbent, add an additional minimum weight on top of the screen to force the swollen absorbent to the bottom and then record the level of the screen. If this test gives a swell of less than 20 mL, repeat using 10 mL of absorbent in the 100-mL graduated cylinder. This empirical test may be replaced by a microscopic calibrated swell of a uniform absorbent particle in the fluid.

11.2.2 Swell may also be checked at 24 h for those specimens that have not shown maximum swell in 2 h. It should be recognized that a 2-h maximum swell may be acceptable but a 24-h swell will probably not be useful for a spill control unit without modification. Such a 24-h swell time may require modification of particle size, etc. to provide effective swell within 2 h. The report shall include the time required to reach maximum absorption if less than 2 h.

11.2.3 Increase in volume of the solid absorbent is the property that distinguishes an absorbent from an adsorbent. Free liquid should be poured out of the graduate cylinder and a weighing then made (with the tared screen). The weight of swollen absorbent allows calculation of absorption efficiency:

$$A = [(W_1 - W_2)/W_1] \times 100 \tag{1}$$

where:

W_1 = weight of absorbed material,
W_2 = weight of unabsorbed material, and
A = absorption efficiency for the fluid and absorbent being tested.

Type I absorbent material may be tested with inert material present such as sand, fibers, etc., that give a special property such as no stir dispersion or sufficient mass to serve as temporary diking. The absorbent mass with inert material can be evaluated using the test described in 8.2. The inert material may be subtracted from the total weights to indicate the absorbent data. The penetration test in 11.4 may also be useful.

11.3 *Rate of Release*—This test can be used to estimate the degree of hazard in the case of flammable or hazardous materials. For those materials with vapor hazards or skin toxicity to humans these are important factors (**warning**—see 10.4.1).

11.3.1 Place a standard sized weighed piece of an absorbent unit in a dish of fluid. Add excess fluid to assure total saturation of material. A saturation time of 30 min should be adequate although a 2-h test is well within the useful practical application time. If material to be absorbed is toxic, run this test in a ventilated fume hood.

11.3.2 Following thorough saturation, hang the absorbent units (minimum 15-cm dimensions) by one corner and weigh immediately (even if dripping). Record the time to cessation of dripping. Reweigh and record the weight as maximum practical pickup rate at the time dripping stops.

11.3.3 Then hang the units in a ventilated hood with the fan on, close the door to obtain maximum air flow (past the units), and continually monitor and record the weight every 10 min. At the end of 2 h (or sooner) if the total weight approaches the tare weight of the unit, record the time and use as a basis for comparison where containment offers an environmental, personal, or flammable hazard. The maximum pick up at the point of no dripping (less than 1 drop per minute) is recorded as the maximum effective pick up of that absorbent unit. This maximum pickup may be recorded as either per unit or per amount of active ingredient in that unit. For given test conditions (temperature, air, flow fluid, etc.), the test will allow selection based on performance.

11.4 *Penetration*:

11.4.1 This test requires a glass tube of 12 to 28 mm inside diameter. Place a porous plug in the bottom of the tube. This can be a cork with a slit to allow air out protected by cloth or tissue paper. Place a 10-cm layer of sand on top of this followed by 15 cm of absorbent material and again 7 cm of sand on top of this. Dye the fluid to be checked and then pour carefully into the top and adjust to maintain a 15-cm head so that when the penetration into the bed is stopped, 15 cm of fluid is above the top layer of sand (held vertically). For safety, place a bottle or container under this apparatus to hold twice the volume of the tube used. This penetration test can be used with pure absorbent as well as those containing inert materials, such as adsorbents, for flow, mass control, etc., with the following change:

11.4.1.1 Compact a layer of sand to 10 cm by dropping a 500-g weight five times for a distance of 3 cm on top of a snug-fitting cover placed above the sand inside the tube; on top of this, place a 15-cm layer of absorbent mixture, compact in the same manner, follow with a 7-cm layer of sand, and compact in the same manner.

11.4.2 Run this test three times for those materials that cause flow to stop within the 15-cm absorbent bed depth and record the average penetration time. Then again record penetration 24 h later. For very volatile materials, a *loose* cork may be placed in the top of the tube to maintain liquid head by minimizing evaporation (this must not form a tight seal).

11.5 *Release to Other Materials*—This test is used to compare the tendency of the absorbent containing fluid sorbate to contaminate either personnel or beaches, for example, through release of contents.

11.5.1 Place a specimen of Type II absorbent (with dimensions and weight recorded) in the fluid to be tested for 30 min. The fluid may be dyed with Oil Red A and should be in a quantity to exceed the capacity of the absorbent unit which means some free fluid will be left after the absorbent unit is removed. Hang the specimen by a corner to allow any adsorbed fluid (as opposed to absorbed fluid) to drain out of fabric, etc. When all dripping has ceased, place the Type II absorbent on a piece of white blotting paper (nominal 3 mm thick) lying on a flat surface. Move the Type II absorbent to a new clean area of blotting paper when the dyed fluid stains through to the reverse side of the blotting paper. When a stain no longer shows through the blotting paper, measure the weight of the absorbent unit. Record this measurement as the *practical containment efficiency.* Repeatedly place this same Type II absorbent specimen on the blotting paper with a 2 g/cm weight uniformly distributed over each square centimetre. Again measure the weight of the Type II absorbent when the stain does not penetrate through the blotting paper. Record this as the *maximum containment efficiency.* The calculation of these two measurements will permit a reliable estimate of the amount of absorbent needed to handle a given quantity.

$$\text{Efficiency} = [(A_f - A)/A_f] \times 100 \tag{2}$$

where:

A_f = weight of absorbent plus fluid, and
A = weight of absorbent.

11.6 *Fluids More Dense than Water* (Sinkers, Bottom Floaters)—This test evaluates the potential to handle materials that sink in water.

11.6.1 Color 1,1,1-trichloroethane (available in many areas as nonflammable spot remover) with a dye such as Oil Red A.

11.6.2 Place water in a vessel to a level of 20 cm. Pour 200 mL of a sample fluid or the actual fluid into the water so it layers on the bottom. Manually hold the Type II absorbent material in the test liquid on the bottom and then retrieve it using a hook and line on a corner. After a maximum pickup time of 30 min, pull the sample up by means of the hook and hold under water for 5 min. Note whether test fluid was released into the water. Then lift out the sample from the water, suspend by the hook, and note dripping of the test fluid, if any. When the drip rate is less than 1 drop every 2 min, weigh the sample. The total amount of fluid absorbed per unit weight of absorbent will indicate the efficiency for underwater pickup as follows:

$$\text{Underwater pickup efficiency} = [(A_f - A)/A_f] \times 100 \tag{3}$$

NOTE 1—For those materials, such as polychlorinated biphenyls, tetraalkyl leads, carbon tetrachloride, ethylene bromide, etc. the rate of release test in 11.5 should also be run and may well be an important consideration. Toxicity and human hazard from running these tests should be given primary and thoughtful consideration. Testing for absorbent Types II and III may be estimated from modules or amounts of Type I absorbent tested. They may be tested as fabricated or assembled, or both, but this testing is optional since the quantities involved will be considerable.

12. Report

12.1 The report shall include the following information:

12.1.1 Test fluid used.

12.1.2 Empirical formula.

12.1.3 Estimated purity (method).

12.1.4 Density.

12.1.5 Boiling point.

12.1.6 Flash point.

12.1.7 Viscosity.

12.1.8 Source.

12.1.9 Absorbent name and whether Type I, II, or III.

12.1.10 Manufacturer of absorbent and estimated date of manufacture.

12.1.11 Date and location of test, including temperature of laboratory and name of person conducting the test.

12.1.12 Indicate whether flammability or toxic hazard exists.

12.2 The report form in Fig. 1 may be useful in recording the results of each test.

13. Precision and Bias

13.1 No precision and bias data are presently available for these methods.

14. Keywords

14.1 absorbents; oil; sorbent; sorbent performance

I. *Conditioning* (Section 9)
  Conditioning temperature ________
  Method of conditioning ________
II. *Oily Material Absorption* (11.2):
  Weight of 2 mL of absorbent ________
  Weight of absorbent and cylinder ________
  Weight of 50 mL of fluid plus absorbent and 100-mL cylinder ________
  Time of stirring before adding screen ________
  Level of mesh screen (If reading is less than 20 mL repeat as below) ________
  Weight of 10 mL of absorbent ________
  Weight of absorbent and 100-mL cylinder ________
  Weight of 50 mL of fluid and absorbent plus 100-mL cylinder ________
  Time of stirring before added screen ________
  Level of mesh screen ________
  Apparent bulk density of absorbent ________
  Total volume of swollen absorbent minus bulk density (volume of absorbent) = the actual increase in volume (pour off liquid) ________
  Absorption efficiency (see 11.2.3) ________
III. *Rate of Release* (see 11.3) ________
  Size and type of absorbent ________
  Weight of absorbent ________
  Weight of active material (if inerts are present) ________
  Weight of absorbent and liquid ________
  Time to stop dripping (minimum ________
  Weight at time of drip cessation ________
  Weight at cessation—weight of absorbent = practical pickup ________
 Practical pickup rate ___ expressed in grams of liquid per grams of absorbent and ___ g of liquid/cm^3 of absorbent ___
 Monitor weight of units in hood air flow

Minutes	A	B	C	D	E	F	G
0							
10							
20							
30							
40							
60							
120							
180							

IV. *Penetration*:	*Test No.* 1 2 3
Diameter of glass tube	________
Length of glass tube	________
Depth of initial penetration bed	________
Depth of penetration after 24 h	________
V. *Release to Other Materials*:	
30-min equilibration fluid:	
Weight of absorbent	________
Absorbent paper used (type and thickness)	
Weight of absorbent when it no longer marks paper	________
Practical containment efficiency (11.5.1)	________
Run test with uniform weight (454 g per each 225 cm^2):	
Weight of absorbent when it no longer marks paper	________
Maximum containment efficiency (for human protection)	________
VI. *Fluids More Dense Than Water*:	
Sinkers	
Weight of 200 mL of fluid	________
Weight of dry absorbent	________
Weight at time of no drip in air for two minutes	________
Underwater pickup efficiency	________

FIG. 1 Sample Form for Recording Data